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VACUUM DEPOSITED JUNCTIONS/

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Submitted by:

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UNIVERSITY OF VIRGINIA  
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## SECTION I

### RESEARCH OBJECTIVES

The principal purpose of this research effort is to investigate the physical electronic properties of thin film junctions which have been produced under very carefully controlled conditions. These junctions are of selected metals, insulators, and semiconductors in order to ascertain what areas of this subject show the most promise in the field of electronic transducer and active device design.

Secondary objectives include the development of the techniques and equipment required for the controlled production of films of preselected thickness and quality.

## SECTION II

### REPORT COVERAGE

This is the first annual status report. It covers the period from December 5, 1962 to December 5, 1963. The first year has been primarily employed to develop the accuracy of the film deposition equipment and to evaluate and calibrate this equipment. This is the principal subject covered in this report.

### SECTION III

#### FILM PREPARATION EQUIPMENT

The film preparation and processing equipment and procedures and the subsequent film tests have been planned in a manner which will afford the maximum control of film dimensions, quality, and test environment. The equipment described is capable of producing a film of any preselected thickness with an indicated accuracy of about  $\pm 5$  percent. The deposition rate may be from 0.1 to 10 angstroms per second.

##### A. General Description

The intrinsic electronic properties of very thin films and film junctions can best be approached by careful and continuous control of the environment which surrounds the films during their deposition and subsequent testing. The films are deposited upon glass and crystalline substrates from molecular oven beams. The substrates are carefully cleaned prior to insertion in the vacuum system and are stripped of sorbed surface layers of gas by flashing to a temperature of approximately 300°C for several minutes. The deposition is carried out in a baked, stainless steel ultra high vacuum system which is electronically pumped (Vac Ion pump) to a pressure of about  $1 \times 10^{-7}$  mmHg (Torr).

The films are deposited from molecular beams which are produced by the evaporation of selected materials from electronically heated crucibles. These molecular beams are columnated and continuously monitored during the deposition cycle. A subminiature ionization gage is located in the plane of the substrate and provides a continuous indication of deposition rate.\* This rate is continuously recorded and also integrated to provide an instantaneous record of the accumulated film thickness. An electromagnetically driven vibrating reed chopper is used to chop that portion of the molecular beam which enters the ionization gage. The chopper produces a 170 cps signal which is amplified by a very narrow band, high gain preamplifier. The signal is then rectified and further amplified by the rate recorder amplifier.

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\* See H. Schwartz, Rev. Sci. Inst., 32, 194-199, 1961.

The present system is provided with three crucibles and a rotatable mask. In this way multimaterial thin film devices may be deposited. Oxygen may be admitted in controlled amounts to permit the production of oxide films. Electrical contacts are mounted on the substrates so that all tests and measurements may be conducted while the film is protected by the vacuum. A future phase of the research will include the investigation of methods of applying protective surface films so that the devices may be safely removed from the vacuum system.

A photograph of the film deposition and processing system is shown in Figure 1. The thin film deposition control console appears at the left. Starting at the bottom of this cabinet the individual items are:

- (1) Vacion pump power supply,
- (2) Feedback controlled d. c. power supply for the filaments which supply the electron emission to heat the boats by electron bombardment,
- (3) Feedback controlled power supply to supply d. c. power for the ion gage filaments,
- (4) Electronic integrator (mounted in table top) to provide an indication of the accumulated film thickness,
- (5) Two dual channel recorders which record film deposition rate, accumulated thickness, system pressure, and substrate temperature,
- (6) Ion gage and substrate outgassing heater controls, recorder event marker controls, film thickness preselector,
- (7) High voltage power supplies for the boat and ion gage plus film rate controls as well as mechanical roughing pump controls.

The vacuum deposition chamber mounted on the top of the 140 liter per second Vacion pump appears in the center of Figure 1. This chamber consists of standard sections of Varian 1 1/2 inch stainless steel ultra high vacuum pipe components. Tees, crosses, and straight sections (all of standard 5 inch length) are employed. This makes an excellent experimental arrangement as different combinations and substitutions are possible which permit additional ports, electrical feed-throughs, and motion

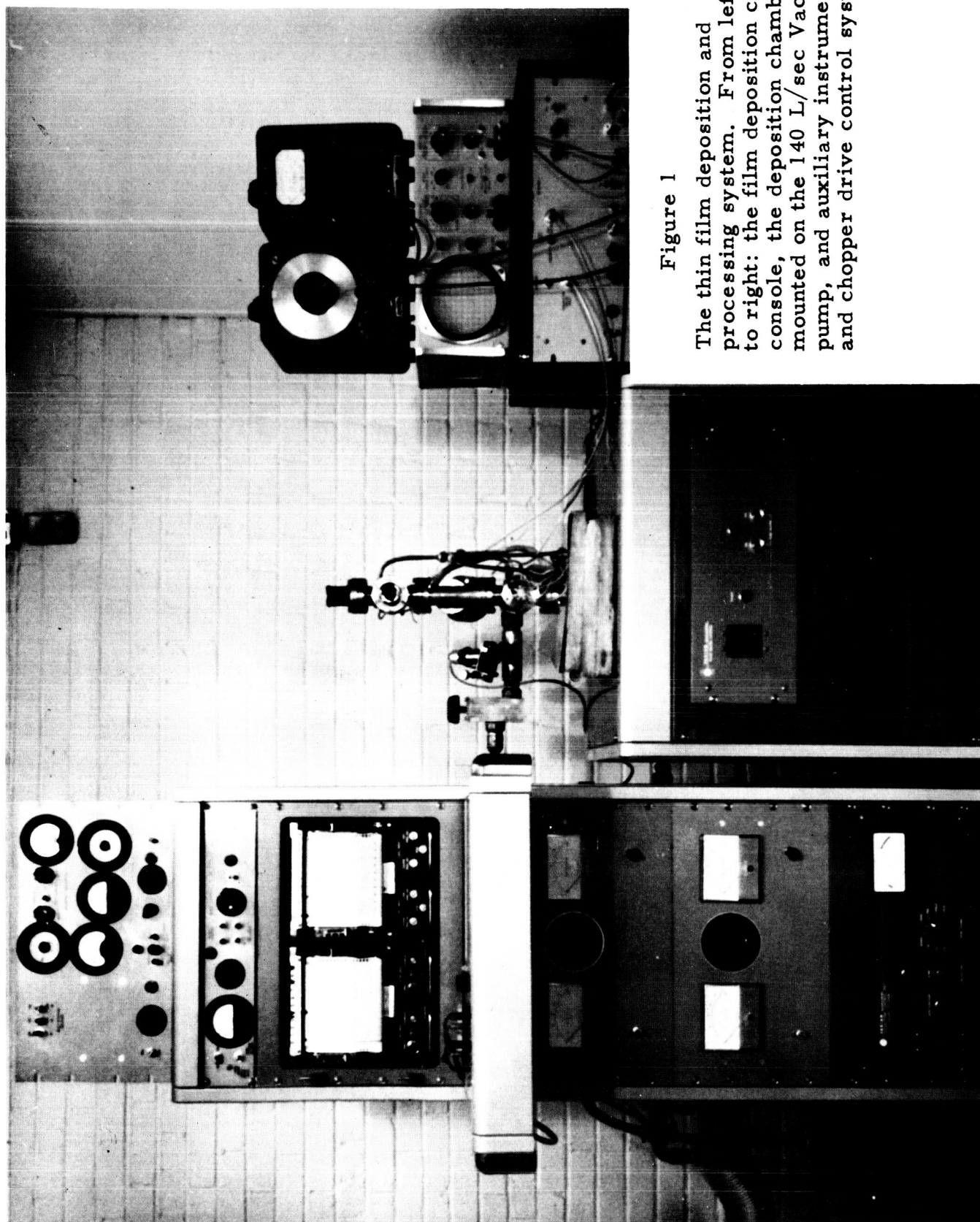


Figure 1

The thin film deposition and processing system. From left to right: the film deposition control console, the deposition chamber mounted on the 140 L/sec Vacion pump, and auxiliary instrumentation and chopper drive control system.

feed-throughs to be moved about and added as needed. All joints are sealed by means of copper gaskets. We have found that by avoiding unnecessary over compression of these gaskets it is possible to use them 4 to 5 times. This plus the small diameter involved results in a system that is very economical to open and reseal. The relatively small size of the vacuum system permits easy bakeout procedures plus good ultra high vacuum pumping characteristics.

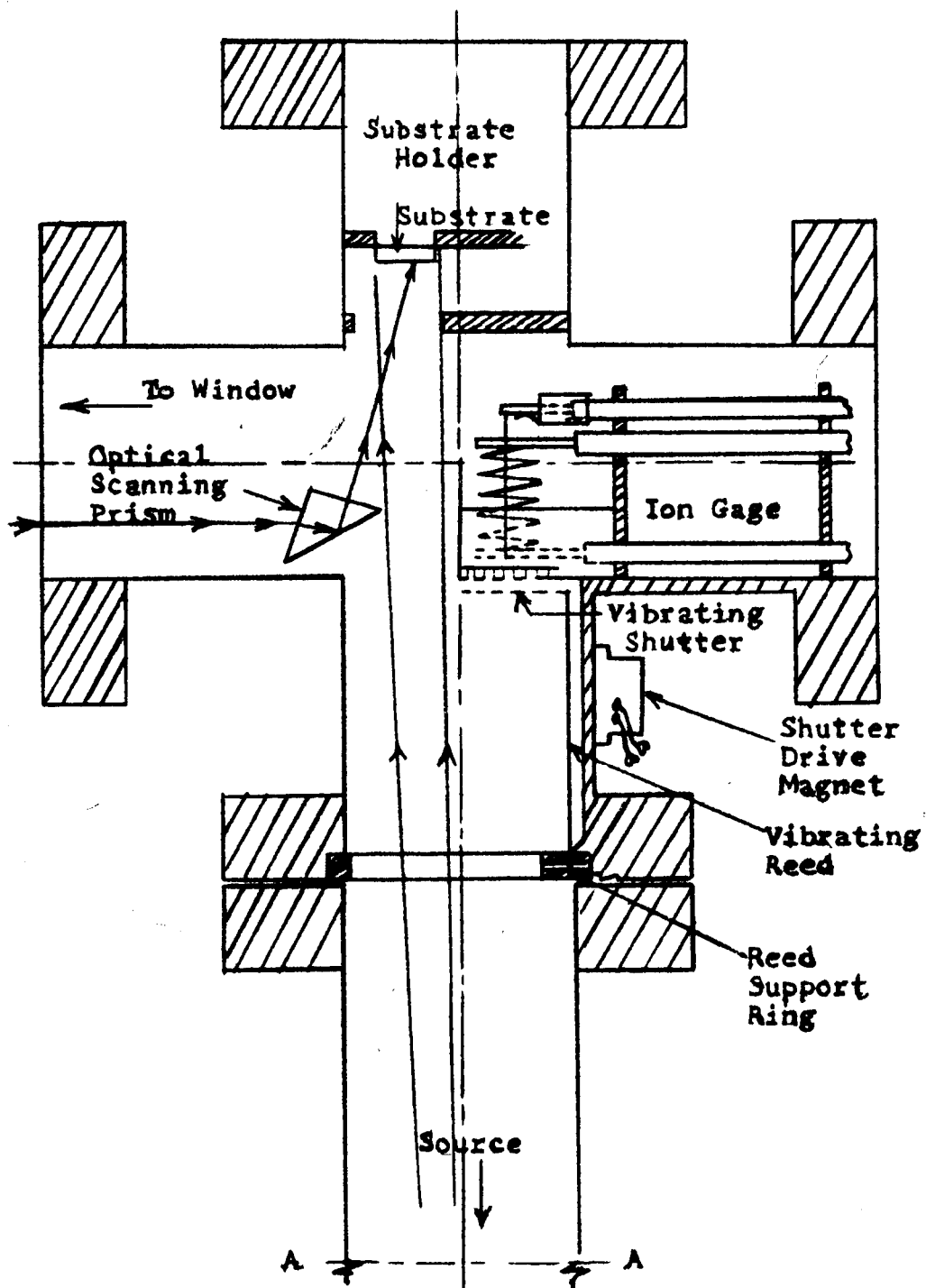
The only change in the vacuum system which does not appear in the drawings is the addition of several turns of 1/4 inch copper tubing on the outside of each section for water cooling purposes. This is especially useful on those sections which house the ion gage and the boat assembly. Future plans call for the location of the boat assembly in a larger chamber which will permit more rapid outgassing of the molten film materials. The chamber will also house a titanium sublimation pump, well shielded from the boats, to provide additional pumping capacity during the evaporation process.

The Vacion pump and associated vacuum system has operated continuously without any leakage problems for one year. The pump is equipped with an internal bakeout heater which is very useful in achieving the  $10^{-9}$  Torr pressure range.

On the extreme right of Figure 1 there appear the auxiliary instrumentation instruments and beneath them the molecular beam reed chopper drive and synchronization control unit. It has been found that only a highly sophisticated electronic drive system would yield the type of chopper response necessary to yield film thickness accuracies on the order of 5 percent. This electronic drive system is described in detail in Section IV.

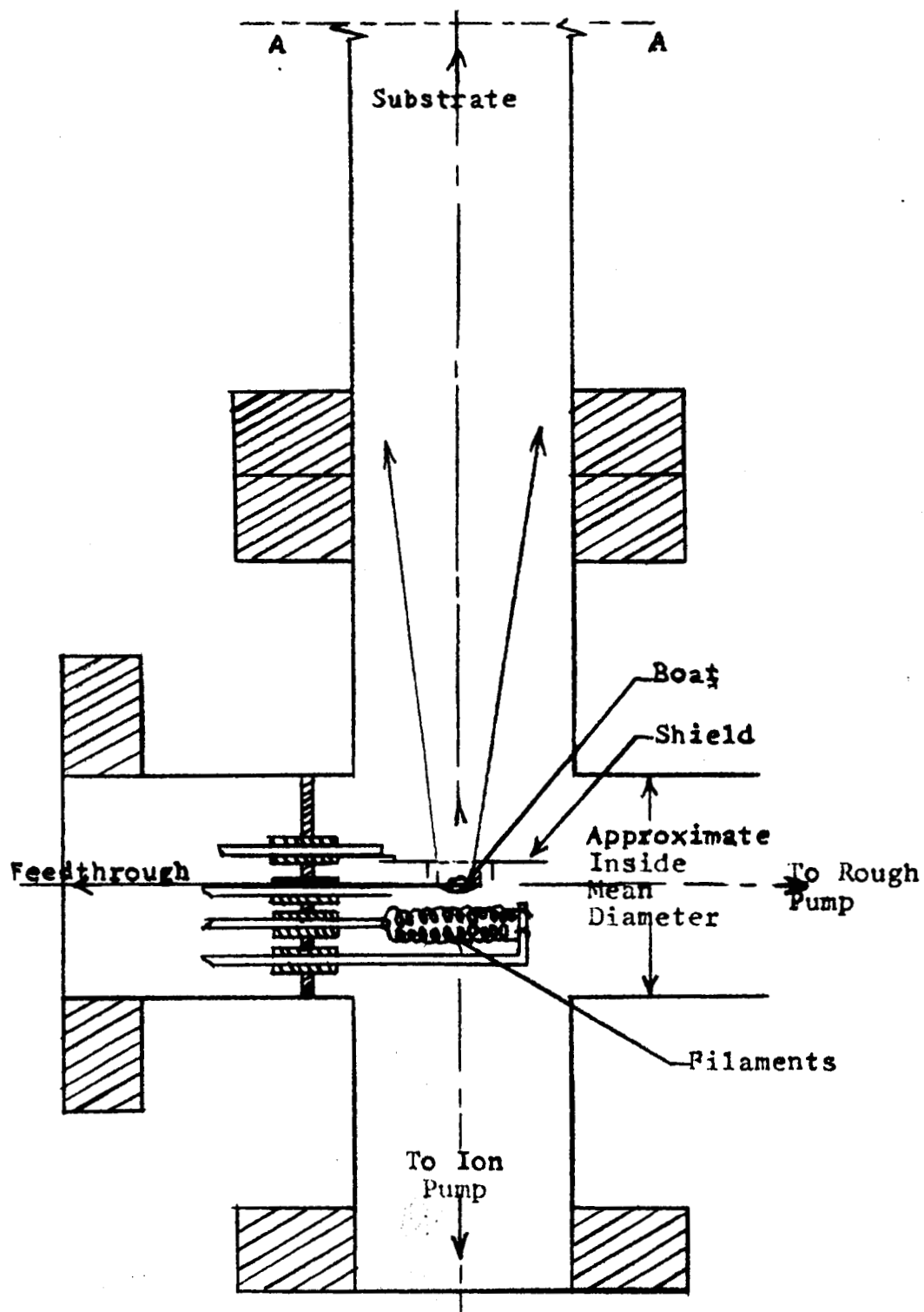
#### B. Deposition Chamber

Figure 2 is a full size sketch of the film deposition chamber. This chamber is a standard Varian 1 1/2 inch ultra high vacuum, stainless steel pipe cross. The flanges are all sealed by means of copper gaskets. This construction is extremely flexible and relatively inexpensive and may be baked at temperatures up to about 500°C provided the electrical



VACUUM SYSTEM VERTICAL SECTION  
Full Scale Sheet 2 of 2

Figure 2A



VACUUM SYSTEM VERTICAL SECTION  
Full Scale Sheet 1 of 2  
Figure 2B

feed-through flanges and the viewing port flange are properly protected according to the manufacturer's recommendations. Low temperature baking (120°C) overnight is equally satisfactory. The electrical feed-throughs provide control and instrumentation continuity. The viewing port plus the associated scanning prism permit viewing the film as well as performing photoelectronic experiments.

#### C. Beam Monitoring Gage

The subminiature ionization gage employed to monitor the molecular beam intensity, and thereby the film deposition rate, is shown in position in Figure 2A. A sketch of this gage also appears in Figure 3 and a photograph of this gage is shown in Figure 8 (c). The gage is of the general subminiature type previously investigated by the author\*. The construction of the gage is similar to the inverted Bayard-Alpert type. The static characteristics of this particular gage are shown in Figure 4. The approximate range of operation of the gage for film deposition rates lying between one tenth and ten angstroms per second lies between  $10^{-6}$  and  $10^{-4}$  Torr.

Repeatability within  $\pm 5$  percent of the deposited film thickness is readily achieved for a given evaporation boat design. It has been found that the molecular optics between the boat, chopper, and ionization gage is very critical and as a result any change in the design of the boat can have a marked effect upon the calibration. Figure 5 displays typical calibration runs for two boat designs. The deposition rate in each case was approximately five angstroms per second.

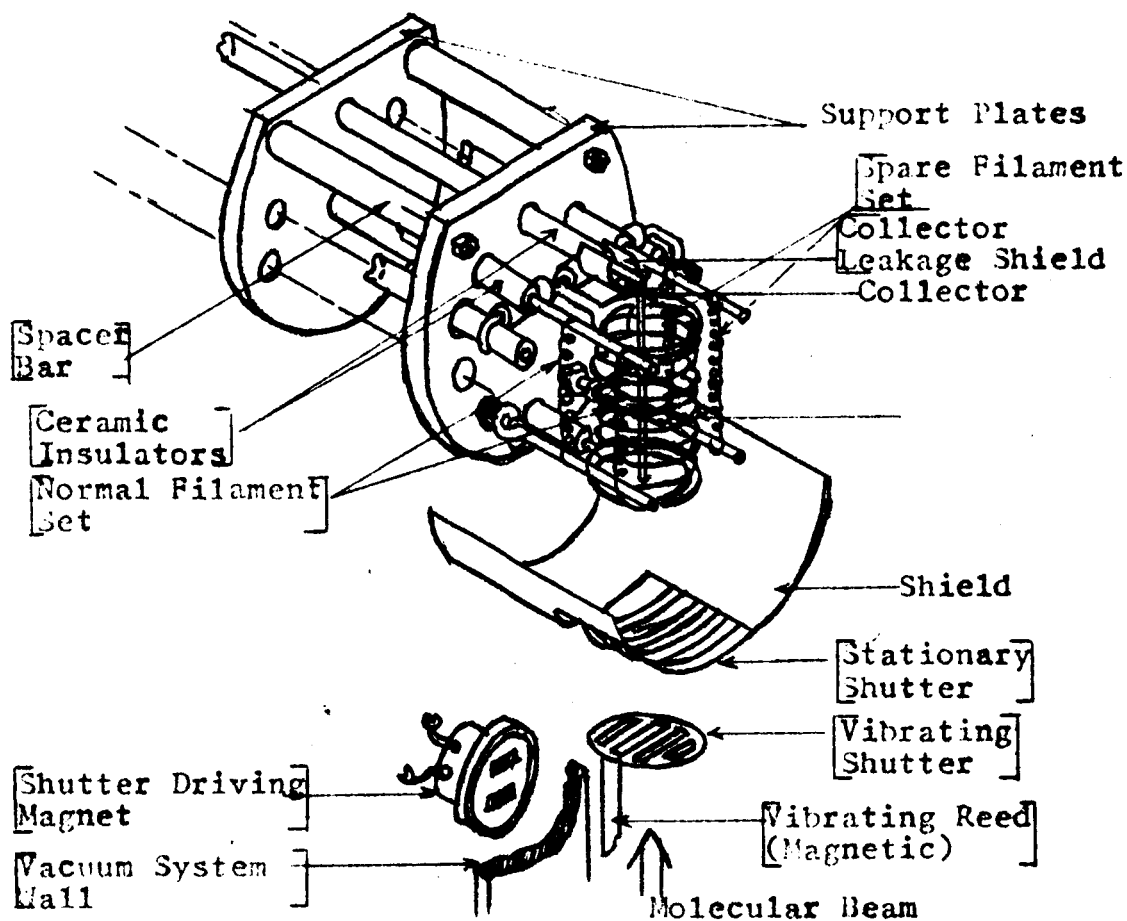
The accumulated film thickness is essentially a logarithmic function of the indicated deposition rate. However, as long as large changes in deposition rate are not permitted throughout a run, the calibration curves shown in Figure 5 are valid.

#### D. Molecular Beam Source

Currently, electronically heated boats or crucibles are used to hold the molten materials to be deposited. Figure 6 is a sketch of the boat

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\* R. L. Ramey, Sixth National Symposium on Vacuum Technology Transactions, p. 85, 1960.



### SUB-MINIATURE IONIZATION GAGE

#### Construction Notes:

1. Accelerator Grid: .365" diam. , .675" long; 10 turns of .010" diam. tungsten wire, closewound on .25" diam. form and allowed to spring to shape.
2. Filaments (each): 20 turns of .006" diam. tungsten wire, closewound on .050" diam. wire form, stretched to about 0.6" under tension; located about 0.1" from grid diam.
3. Collector: 0.010" diam. tungsten wire, 0.675" long .
4. Leads (8): 0.050" diam. nickel alloy, furnished with feedthrough.
5. Vibrating Shutter: 0.001" molybdenum with 0.050" slots spaced 0.050" apart.
6. Vibrating Reed: From 60 cps frequency meter, steel, magnetic.

**Figure 3**

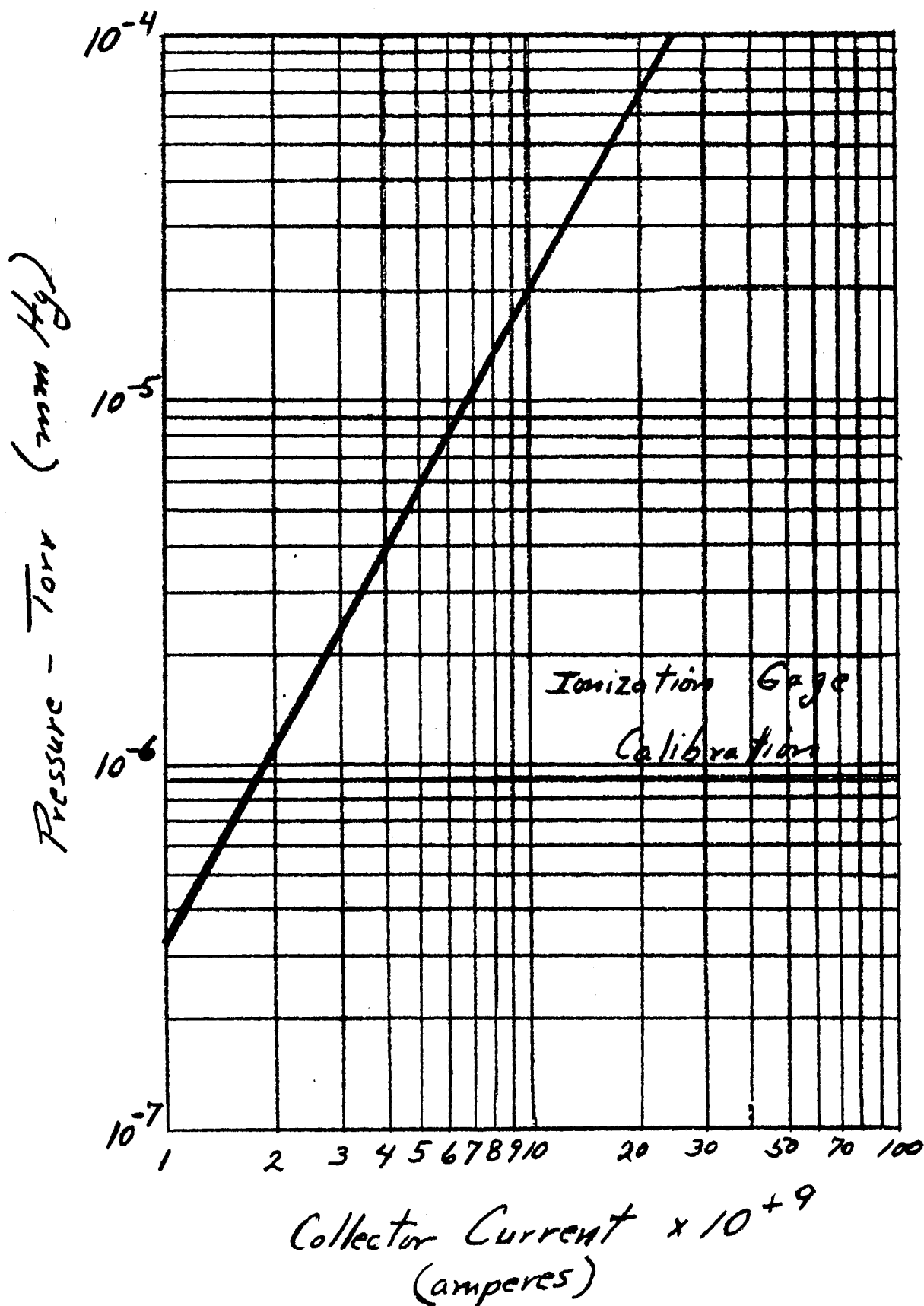


Figure 4

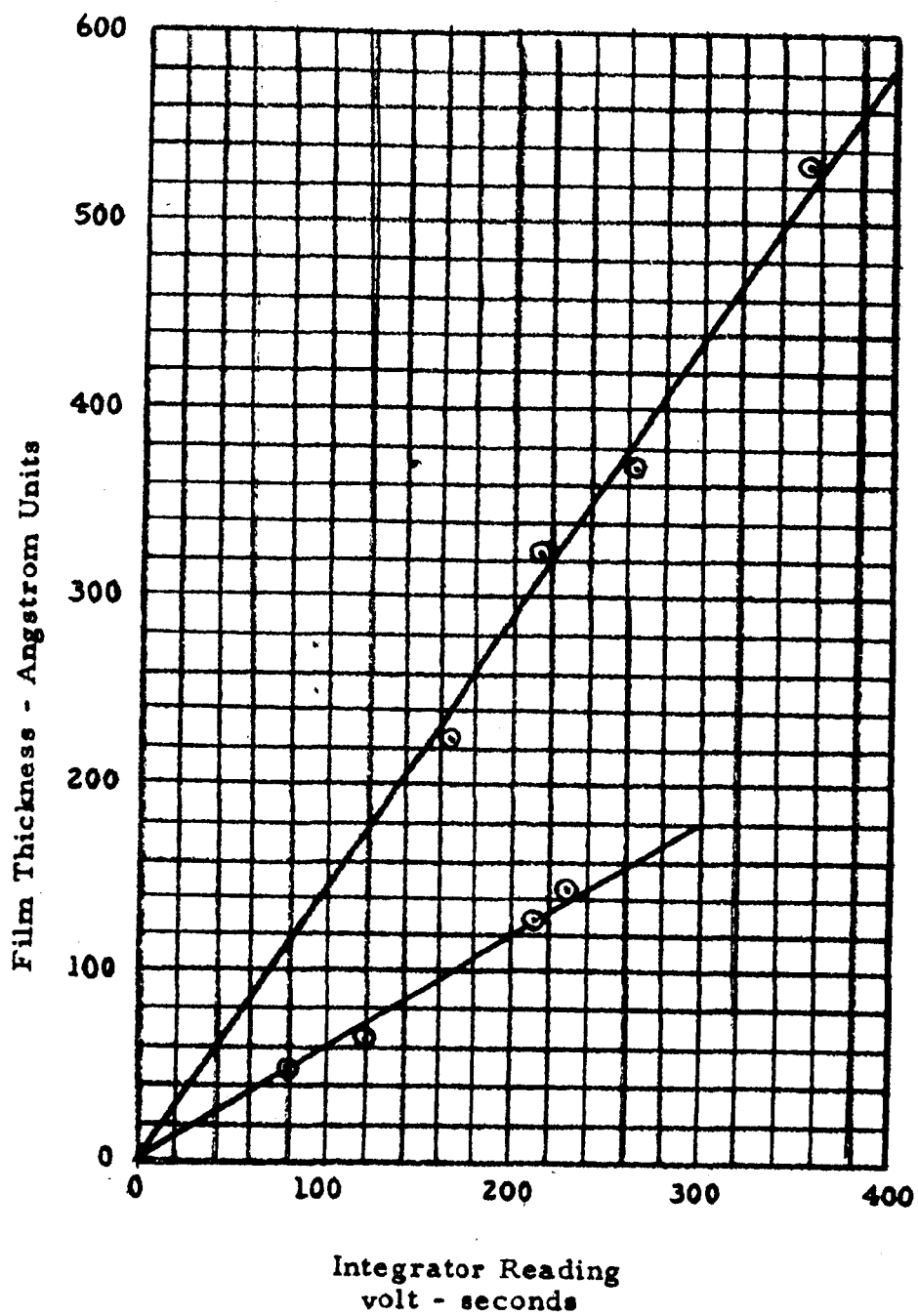
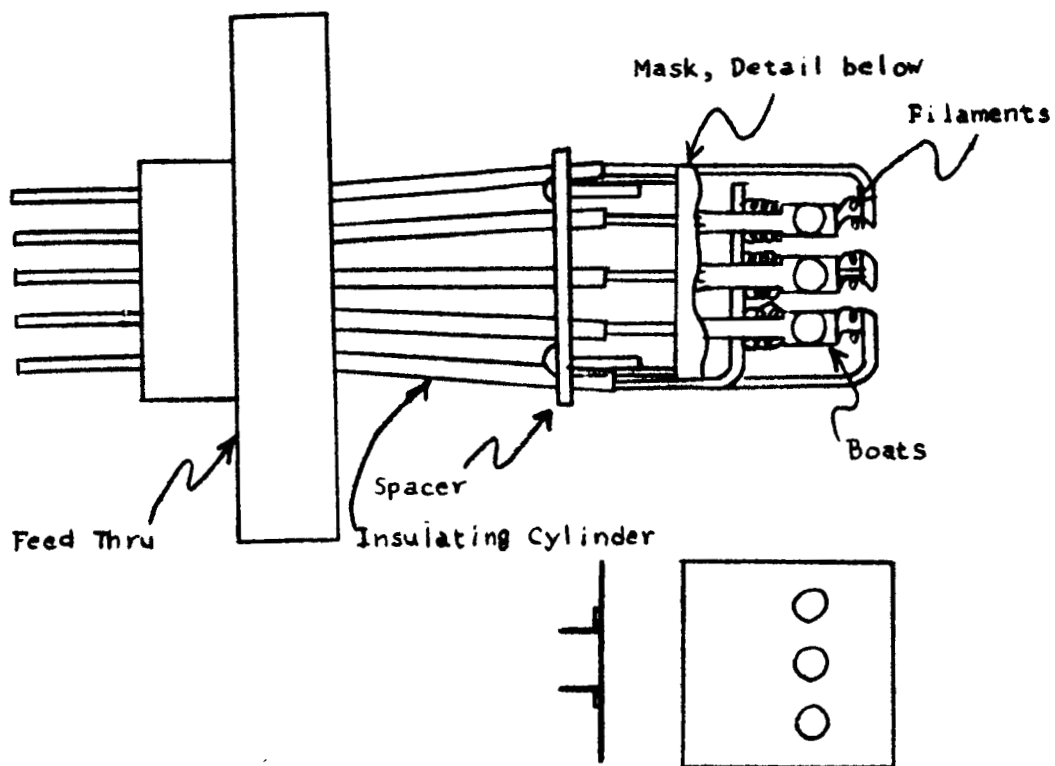
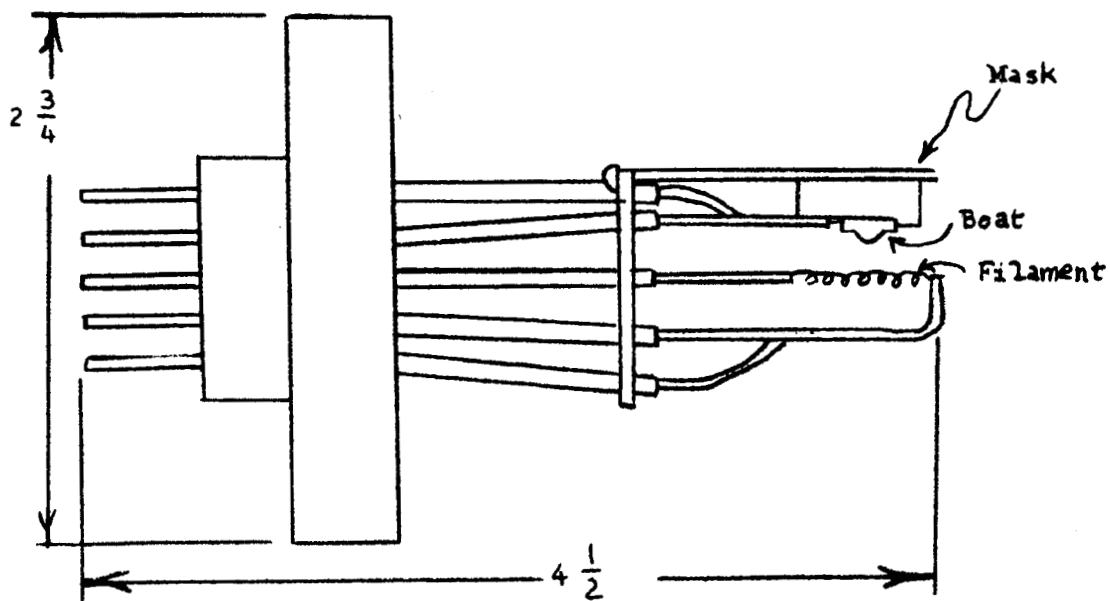


Figure 5. Typical calibration curves for a deposition rate of approximately five angstroms per second.



Mask Detail



Boat Assembly

Figure 6

assembly and a photograph of a typical unit is shown in Figure 8 (b). Molybdenum and tantalum boats are normally used and tungsten boats may be substituted. The boat to substrate spacing is ten inches.

Excellent heating control plus long boat life is achieved by the use of electron bombardment heating of the boats. Each boat functions as an anode and is provided with a filamentary cathode. The power consumption usually runs between 10 and 40 watts for charges on the order of one or two hundred milligrams. It is important that the boat filament be shielded from the substrate to prevent tungsten contamination.

The crucible temperature is established by automatic control of the cathode emission current. Operation is normally in the saturation emission current range. It has been noticed that maximum power transfer to the anode is sometimes voltage dependent for certain boat assemblies. This is believed to indicate that a fraction of the emission current is bombarding parts of the anode structure other than the crucible itself with an attendant reduction in effective heating power. The emission current is obtained from a special constant current regulated power supply.

It is possible to completely close a feedback loop and place the system in automatic deposition rate control. However the thermal time lags in both the filament and the crucible require that the time lag in the feedback loop be approximately one second or longer. As a result, experience has shown that manual operation of the rate control is completely adequate for the deposition times currently encountered.

The question of crucible contamination of the film is an ever present problem and must be given consideration. Minimum contamination is achieved by selecting film material - crucible combinations where the degree of alloying is very small. For instance, visual examination of a crucible which has been fully emptied by evaporation should not show any signs of alloying. Alloying can essentially be eliminated by holding the crucible at room temperature while the film material is heated by means of electron bombardment. The crucible is usually designed as a water cooled heat sink in the smaller units. A magnetically

bent electron beam is used to directly heat the material to be evaporated. The bent magnetic beam is preferred as it permits the beam's cathode to be hidden from the crucible and in this manner filament contamination may be avoided. Future plans will undoubtedly include the incorporation of such a crucible. Commercial units for this purpose have undergone extensive development during the past two years and compact designs are currently appearing on the market.

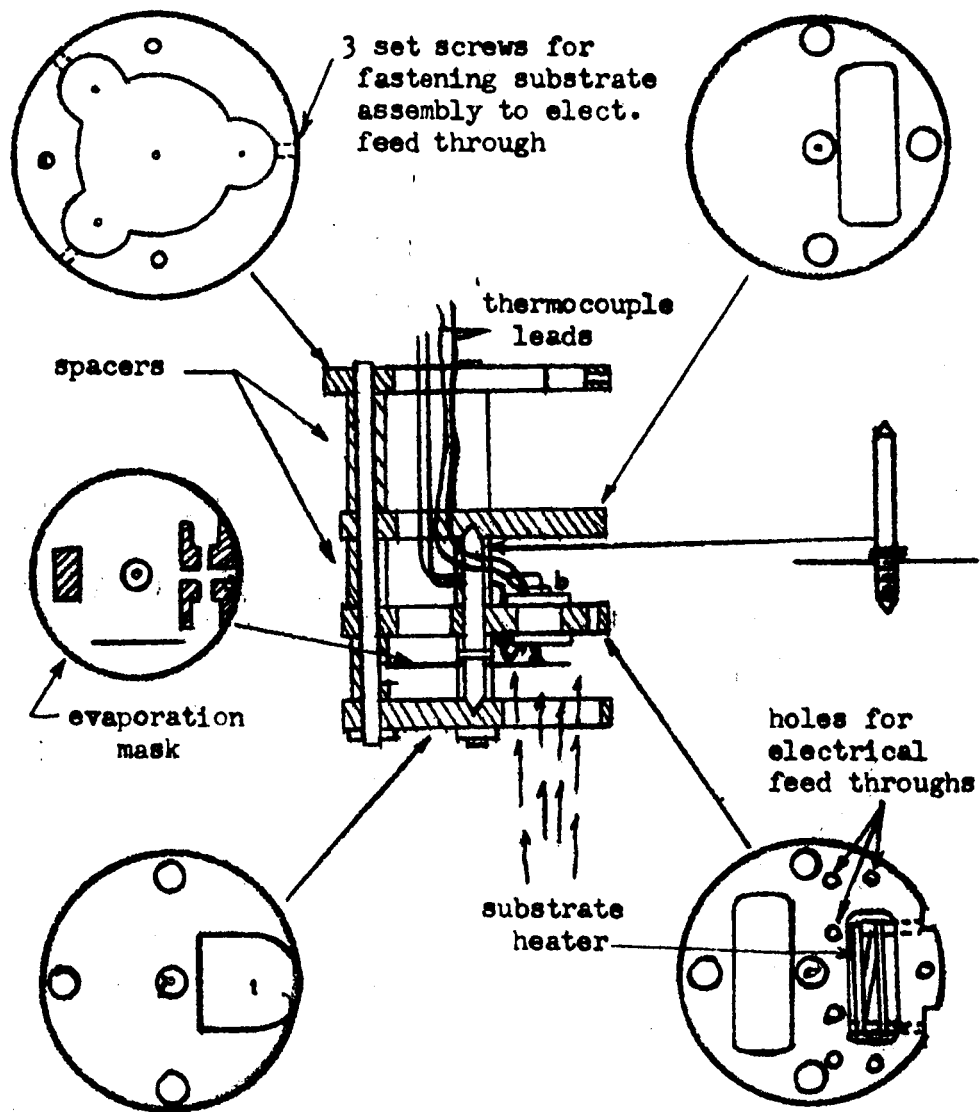
#### E. Substrate Holder

The substrate holder serves two primary purposes: to hold the substrate behind a rotatable multimask and to provide the proper substrate temperatures for surface flashing and subsequent film deposition. Figure 7 is a sketch of the substrate holder and a photograph of this unit appears in Figure 8(a). Two identical substrates are used. One substrate faces the beam while the other faces away from the beam. The substrate heater (tungsten ribbon) is mounted between the substrates. A mean substrate surface temperature is obtained by monitoring the surface temperature of the rear or mock substrate. A platinum/platinum-13% rhodium thermocouple is used to measure the mock substrate surface temperature.

The substrate may be glass, metal, or any suitable signal crystal material. Currently glass and aluminum foil substrates are being used during the testing and calibration of the equipment. Aluminum foil is preferred because a Cahn micro-balance is being used to determine the weight of the deposited film during the calibration tests and it is important to keep the tare weight as small as possible so that the maximum sensitivity of the balance may be realized.

The evaporation mask may accommodate up to 5 positions. It is rotated by means of an external magnet and a soft iron yoke which is clamped to the mask. The mask is readily changed. The only precaution that must be observed with a rotation mask which carries more than two mask positions is the problem of accurate indexing. This is difficult to achieve and only by planning of the mask layout for radial positioning may accurate one-dimensional positioning be achieved. To avoid this

# SUBSTRATE ASSEMBLY



a - probe to gold contacts on substrate

b - thermocouple sealed in glass for measuring temperature of substrate

FULL SCALE:

Figure 7

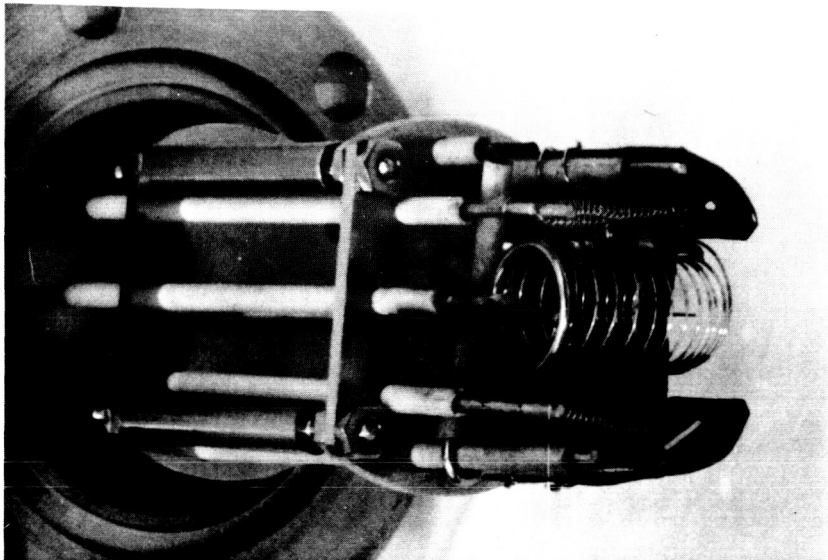
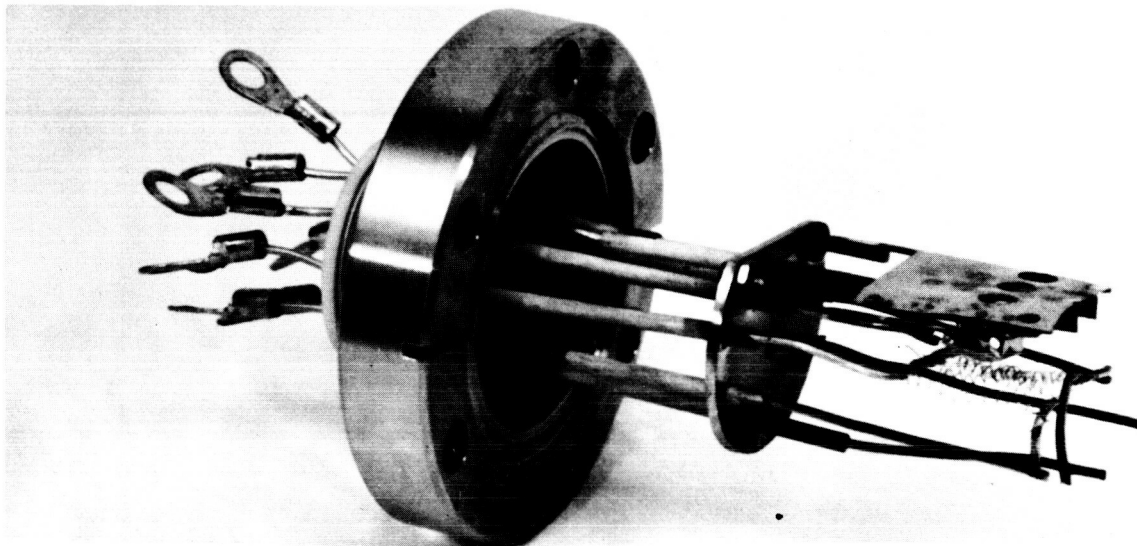
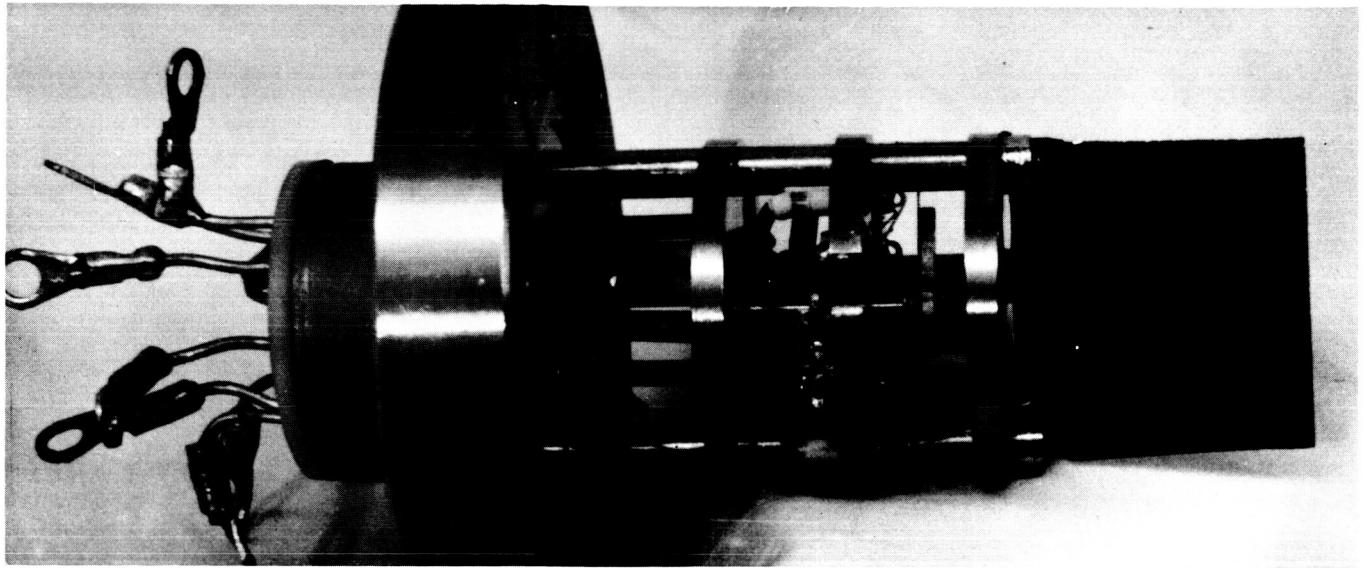


Figure 8

The major components of the vacuum chamber:  
 (a) the substrate holder with rotatable mask;  
 (b) a triple crucible, electronically heated;  
 (c) the miniature ionization gage.

problem a linear motion feedthrough (bellows sealed) has been obtained for use on a mask holder for the thin film junctions. This will permit us to achieve two dimensional positioning accuracy with the masks and will replace the rotary mask assembly.

Gold contacts are placed upon the substrate in a separate Vac Ion pumped deposition system. Tungsten probes make electrical contact with the gold contacts when the substrate is placed in the substrate holder. The deposited films overlap the gold contacts and in this manner reliable electrical connections are made to the film during processing and for later electronic testing.

The metal shield which appears at the right-hand end of the substrate holder is used to shield the substrate from the ionization gage filament and hence possible tungsten contamination.

## SECTION IV

### FILM PREPARATION CONTROL INSTRUMENTATION

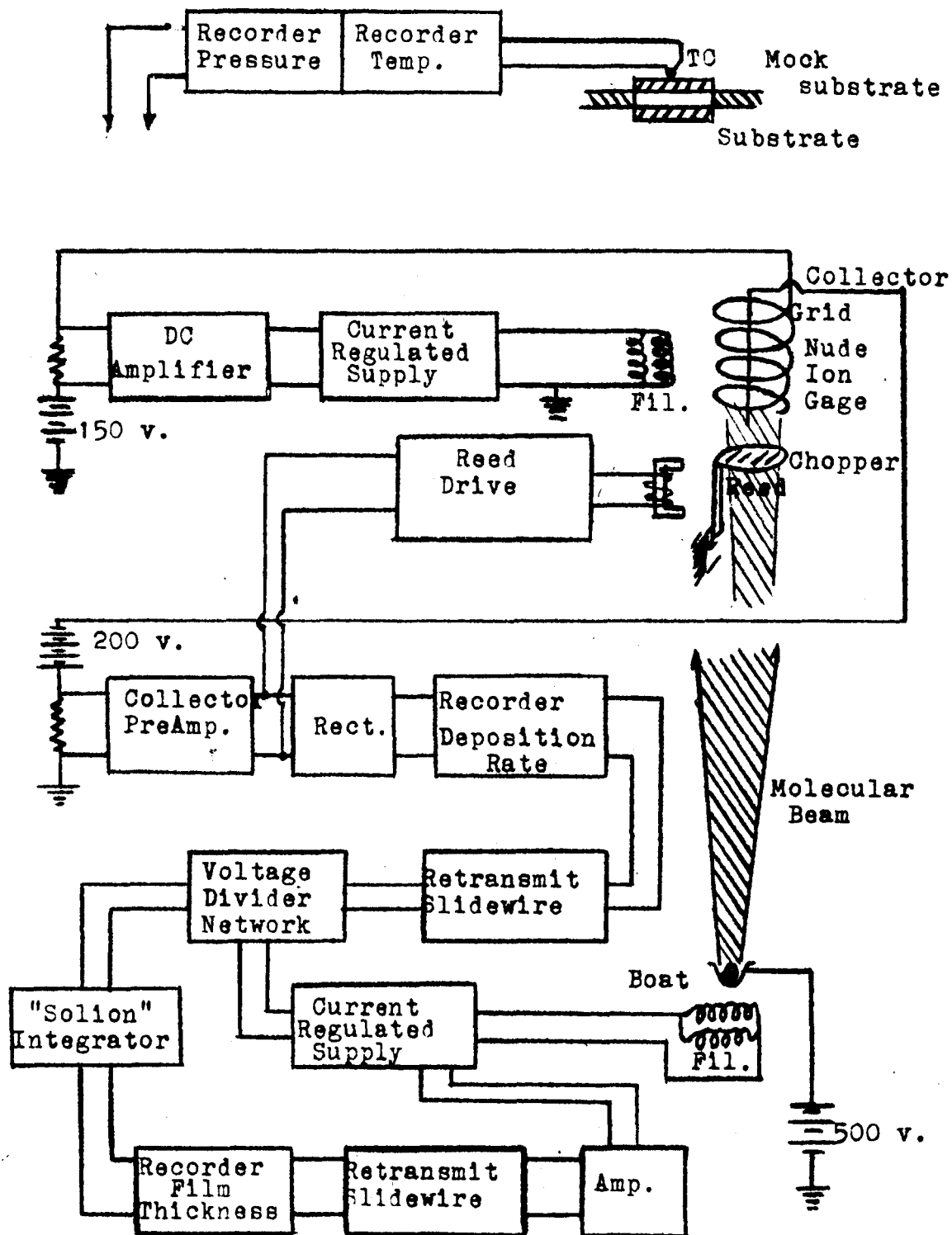
Figure 9 is a block diagram of the film deposition control system. The substrate and mock substrate for temperature monitoring appear at the top of the sketch. The surface temperature of the mock substrate is continuously recorded and this information may be used to automatically control the substrate temperature if this should become desirable. In some instances it is desirable to record the resistance of the film as it is deposited. This may be achieved by means of a general purpose recorder channel which is available for other testing as the need arises.

#### A. Ionization Gage Emission Regulator

The ionization gage control system maintains constant electron emission from the filament of this gage. This is accomplished by electronically monitoring the accelerator grid current. This circuit may be traced in Figure 10. The ion gage accelerator grid is connected to the 150 volt d. c. grid power supply and then through potentiometer R2 to ground. A voltage proportional to the grid current is developed across this potentiometer and it introduces a signal current in the transistor, TR3, in the ionization gage regulation feedback circuit. An increasing grid current will introduce an increasing base and hence collector current in this NPN transistor. As the collector voltage varies, the current through the time delay circuit R7C3 to the ion gage filament supply is varied. This current increases with increasing accelerator grid current.

This control current is fed into a differential amplifier (TR10 and TR11) where an increasing grid current results in a decreasing base current to TR9. This in turn reduces the base current to TR8 which controls the series regulator (TR4, 5, 6, 7 in parallel) and reduces the current output of the ionization gage filament power supply.

The ionization gage filament power supply is also self-regulating if the ionization gage regulator should be disabled. This is accomplished by means of the voltage drop developed across R23 which is in series with the negative output lead of this power supply. A current proportional



THIN FILM DEPOSITION CONTROL SYSTEM

Figure 9

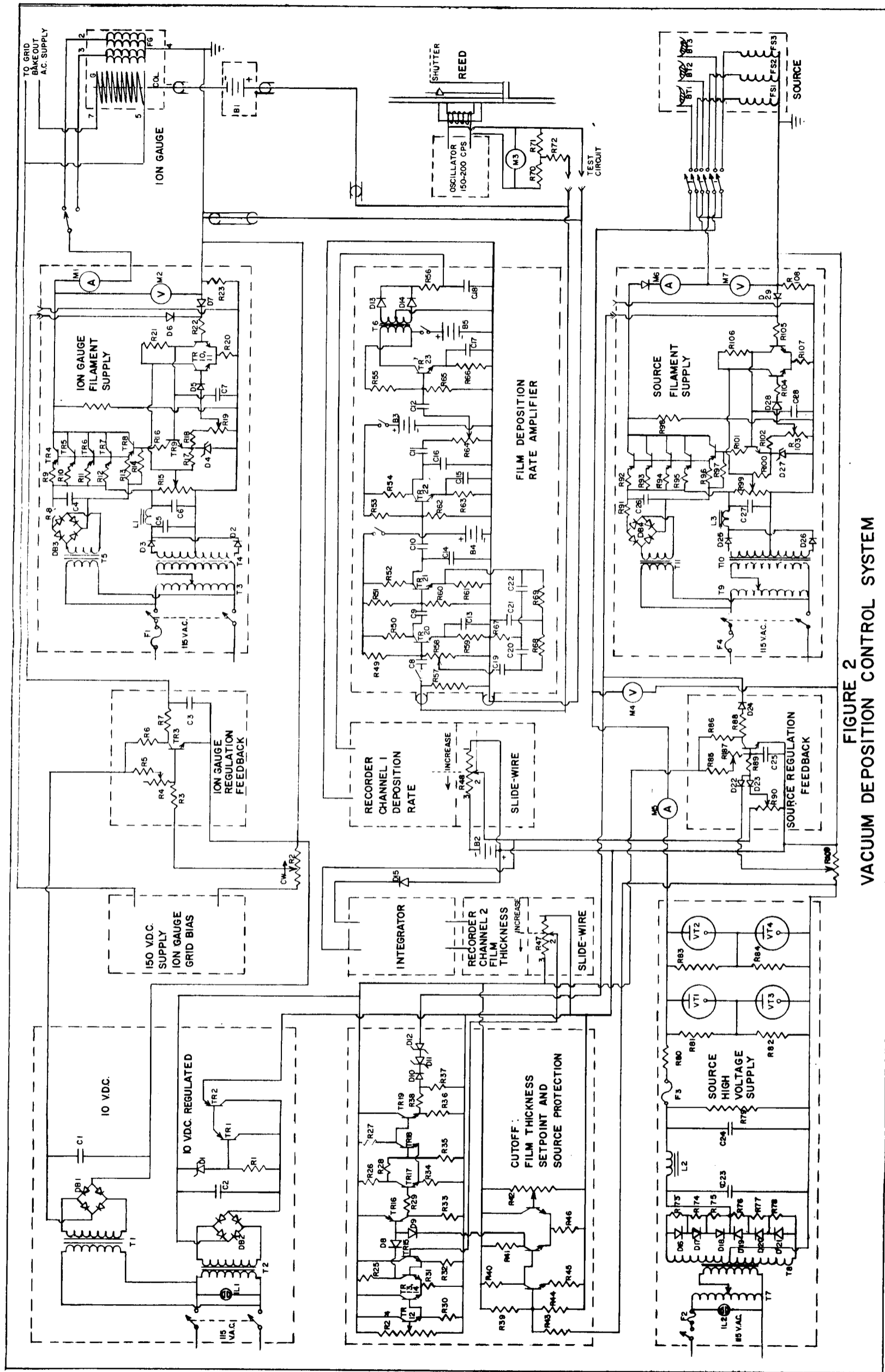


FIGURE 2  
VACUUM DEPOSITION CONTROL SYSTEM

to the current through R23 is fed into the differential amplifier. By adjusting the setting of R19 the maximum output current of the power supply may be selected. Then any feedback current from the ionization gage regulator will tend to reduce the output of this power supply from the maximum value to the proper value for the desired emission current.

The main rectifier in the ionization gage power supply consists of D2 and D3 plus a pi-section filter C5-L1-C6. The auxiliary power supply (bridge rectifier DB3) is used to furnish a current to cancel  $I_{CO}$  which arises from TR4, 5, 6, 7 and 8.

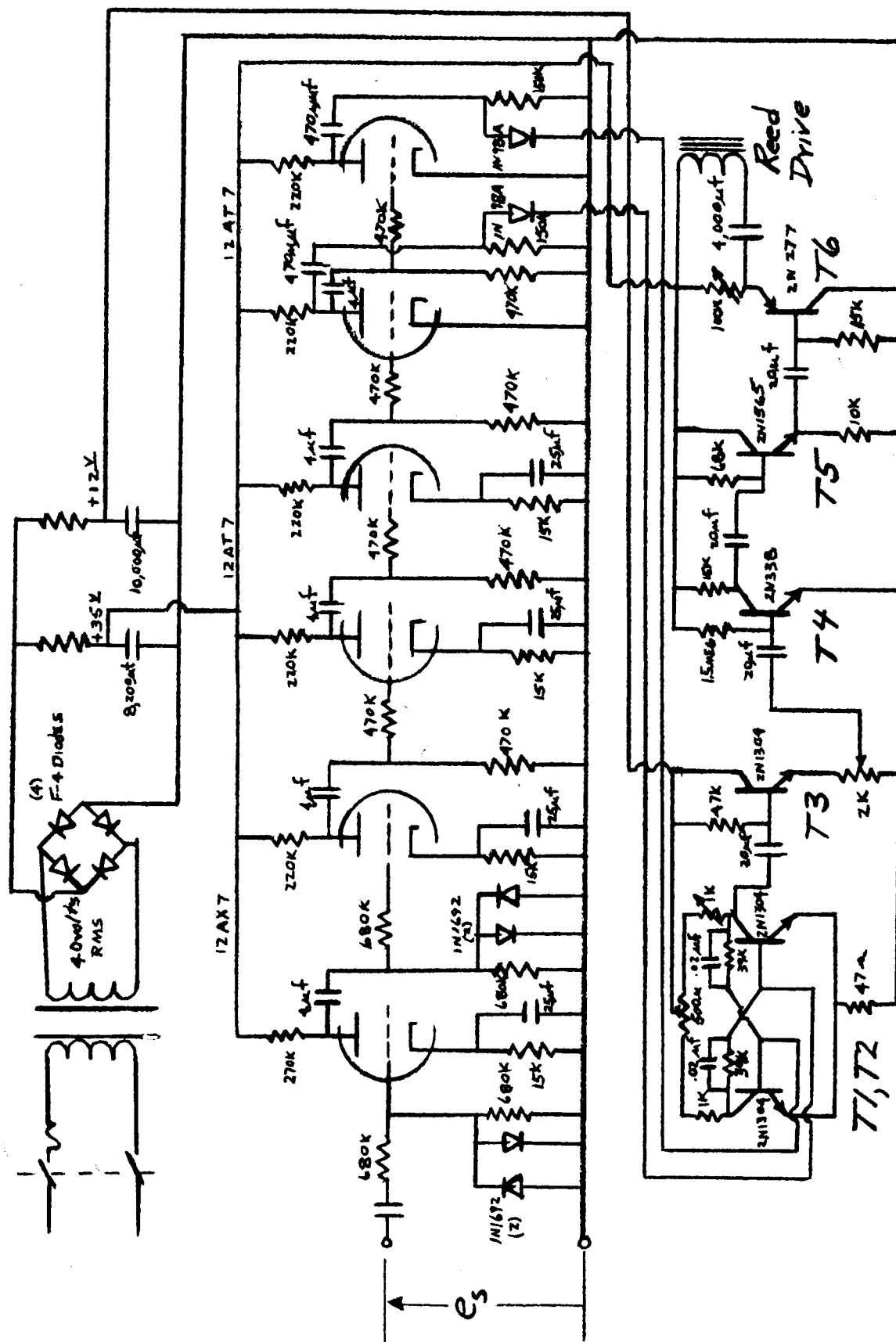
#### B. Molecular Beam Modulator

The third item from the top in Figure 9 is the molecular beam modulator. Modulation is accomplished by means of a vibrating reed shutter which produces a 170 cps sinusoidal signal at the collector of the ionization gage. A frequency drift of approximately 1 cps is encountered during deposition. This frequency variation is sufficient to noticeably alter the reed response and hence the amplitude of the signal as measured at the ionization gage collector.

To solve this problem it was found necessary to design a special oscillator which will start the reed vibrating. As soon as a signal appears at the output of the ionization gage preamplifier the oscillator is electronically locked to this signal. The vibrating reed actually becomes the frequency determining element of a feedback oscillator.

The reed drive circuit is shown in Figure 11. The multivibrator consists of transistors T1 and T2. This circuit is adjusted to the exact drive frequency for the reed at the beginning of a run. Although the waveform is a square wave the reed response is very sharp. Transistor T3 constitutes an emitter follower, buffer amplifier; transistors T4 and T5 are additional current amplifiers. The power output stage, T6, drives the reed.

Synchronization of the reed frequency to the ionization gage pre-amplifier output,  $e_s$ , is accomplished by means of six squaring and amplifying circuits. This produces a constant amplitude square wave



from the sinusoidal preamplifier signal. This square wave is then differentiated and the positive pulses obtained are used to synchronize the multivibrator on each half cycle.

#### C. Ionization Gage Preamplifier

The next item in Figure 9 is the ionization gage preamplifier. The circuit appears in the right center of Figure 10 under the heading "film deposition rate amplifier." This is a four stage amplifier with a transfer impedance of approximately  $10^9$ .

A feedback circuit is placed around the first two stages. A twin-T notch filter is placed in the feedback loop. The center frequency of the notch is about 170 cps (reed frequency) and the stop-bandwidth is about 15 cps. As a result the feedback is very large for all frequencies except those blocked by the notch filter and therefore the pass band of the amplifier is equal to the stop band of the notch filter. In this manner a narrow band amplifier of very high gain and relatively low noise is available to amplify the collector current of the ionization gage.

The output of this preamplifier is fed to the reed drive circuit (Figure 11) as an a. c. signal to be used in synchronizing the reed drive oscillator to the modulated signal. The output of the preamplifier is also rectified and fed to the d. c. recorder amplifier where it is recorded as film deposition rate.

#### D. Film Thickness Computer

The film rate recorder pen drives a retransmitting slidewire. A bias voltage is applied to this slidewire potentiometer, the output of which is fed to an electrochemical integrator (Solion). The output of the integrator is proportional to accumulated film thickness and is recorded on another recorder channel.

The accumulated film thickness recorder pen also drives a retransmitting slidewire. The output of this biased potentiometer is indicative of the accumulated film thickness and is fed through an emitter follower buffer amplifier TR13, 14 (See "Film thickness setpoint and source protection" circuit, left center, Figure 10). The d. c. signal from the film thickness retransmitting slidewire is compared to a preselected

signal from the film thickness setpoint potentiometer, R24.

When the accumulated film thickness equals the preset value, a decreasing voltage is obtained from the differential amplifier. This signal is amplified and is then used to trigger a Schmitt trigger circuit (TR17, 18). The output of this amplifier is a positive going step function which is coupled to the boat filament power supply through the Zener diodes D11 and 12. These diodes are used to adjust the voltage levels between the circuits involved.

Excessive source filament emission current is prevented by comparing the emission current through R109 to a preselected current from R42. The result is fed into the Schmitt trigger circuit to cut off the filament power supply if a preset level is exceeded.

#### E. Source Filament Supply

The filament supply for the individual boats is essentially identical to the feedback controlled power supply that is used for the ionization gage. This power supply was discussed in detail in part (A) of this section and therefore will not be discussed further.

A maximum current output level is selected by means of the voltage developed across R108. The cutoff signal from the film thickness setpoint circuit (part D) is fed into the differential amplifier through resistor R105. Evaporation rate is controlled by feeding a control signal into this differential amplifier at the same point.

#### F. Source Regulation

By controlling the emission current of the boat filament, it is possible to control the temperature and thereby the evaporation rate from the boat. The boat-to-filament accelerating potential is supplied by the high voltage power supply (lower left-hand corner of Figure 10).

The source regulation feedback circuit (lower center of Figure 10) is designed to operate from two possible signal sources. Potentiometer R109 provides a signal which is proportional to the emission current. This signal is amplified and fed into the differential control amplifier of the source filament supply. Manual control of the evaporation rate is achieved by adjustment of R109.

Automatic rate control may be achieved by means of a rate signal obtained from the retransmitting slidewire of the rate recorder channel. Because of the thermal time lags involved in this feedback loop (about 1 to 2 seconds) it has been found that manual rate control is often more satisfactory.

## SECTION V

### STATUS OF PROJECT AND PROJECTED PLANS

The thin film deposition equipment has been designed, constructed, and tested. It is now possible to proceed with the preparation of films of preselected average thickness. Controlled processing and testing conditions exist in the vacuum systems.

Current plans call for investigation in three basic areas of thin film phenomena: conductivity, Hall effect, and photoelectric effects. This will include both single films and rectifying junctions between dissimilar films.

Plans have been made to include a study of several thin film devices at the same time the basic studies are in progress. These devices will probably include the field effect transistor, thin film magnetometer, and photo-diodes.

Currently, three candidates for the masters degree and two candidates for the doctorate are active on this project.

The results of the development of the film processing equipment described in this report are being prepared for publication subject to the approval of the sponsor.

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